Abstract No. Rave0208

Oxidation of Iron in Gold/Iron Core/Shell Nanoparticles

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Beamline(s): X11A and X23B

Introduction: Recently, reverse micelles were used to produce core/shell nanoparticles of iron and gold of highly uniform size distribution and reported to contain metallic iron. A composite structure of this sort is attractive in biosensor and bioactuator applications; the metallic iron core provides high magnetic sensitivity and the gold coating is easily functionalized. With iron/gold nanoparticles prepared by this method, we directly measure the chemical state of the iron component using X-ray Absorption Spectroscopy (XAS) and interpret our results along with the measured magnetization of the particles. In earlier reports, the metalicity of the iron core was inferred from magnetization measurements rather than directly measured. In contrast, XAS directly measures of the structural environment of the iron atoms. Because XAS interpretation is independent of symmetry or periodicity, it is an ideal tool for characterizing nanoparticles. Using XAS, we demonstrate that the iron component of the iron/gold nanoparticles is fully oxidized. We suggest that oxidation is a persistent problem in core/shell nanoparticles of this variety and recommend that characterization by XAS be a standard part of nanoparticle preparation.

Methods and Materials: Iron(II) chloride contained in the aqueous core of a reverse micelle was reduced to iron metal using sodium borohydride and made to cover a gold core previously prepared in the micelle. The iron coated core then served as a nucleation source for a gold shell formed from the reduction of hydrogen tetrachloroaurate. The micelle solution was formed using the surfactant system of cetyltrimethylammonium bromide, octane, and *n*-butanol as a co-surfactant. Samples for XAS measurements were prepared by spreading a quantity of the dried nanoparticles uniformly over pieces of cellophane tape. Data were collected at beamlines X11a and X23b.

Conclusions: Oxidation appears to be a severe problem in this reverse micelle method of nanoparticle production. Indeed, we suggest that oxidation may be a problem endemic to all forms of production of nano-scale iron particles. Because passivated magnetic nanoparticles are of such significant technological interest as components of sensors and actuators requiring small particles with high magnetic susceptibilities, controlling the oxidation of the iron component is vital. We have shown that magnetization is a poor technique for determining the iron species due to its considerable uncertainty. We suggest that X-ray Absorption Spectroscopy be used as a standard part of nanoparticle characterization in any situation where oxidation is a concern.

Acknowledgments: This work was supported in part by the American Society of Electrical Engineers. We thank J. ~Byers, M. ~Raphael, and M. ~Willard for helpful discussion.